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(PATENT)

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant: V. Chan et al.
Serial No.: 09/720710
Confirmation No.: 5662
Filed: July 2, 1999
For: PERIODIC POROUS AND RELIEF NANOSTRUCTURED
ARTICLES
Examiner: Chang, Victor S.
Art Unit: 1771

DECLARATION UNDER 37 C.F.R. §1.132

I, Professor Edwin L. Thomas, declare to my own knowledge that:

1. I am a Professor of Materials Science and Engineering at the Massachusetts Institute of Technology (MIT). I am also Head of the Department of Materials Science and Engineering at MIT. I was the Founding Director of the MIT Institute for Soldier Nanotechnologies from 2002-2006, a center to enhance soldier survivability and protection by developing nanoscience and nanoengineering concepts. I served as Associate Head for the Department of Materials Science and Engineering in 1995-1996, and, from 1989 to 1995, served as the Director in the Program in Polymer Science and Technology at MIT. From 1985-1988, I was Head of the Department of Polymer Science and Engineering at University of Massachusetts, where I founded and served as Co-Director of the Institute for Interface Science from 1986-1989.

I have been a Visiting Professor at Caltech in May, 2005, Visiting Professor/Senior Scientist at the Institut Charles Sadron at the CNRS for Macromolecules in Strasbourg, France in 1997, Visiting Professor in the Department of Physics at Bristol University in 1985, a Bye Fellow at Robinson College, in the Department of Physics and Materials Science at Cambridge University in 1984, a Visiting Professor in the Department of Chemical Engineering and Materials Science at the University of Minnesota in 1983, the Alexander von Humboldt Fellow at the Institute for Macromolecular Chemistry at the University of Freiburg in 1981, Assistant

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Professor in the Department of Chemical Engineering and Materials Science at the University of Minnesota from 1973-77, and research assistant at the Federal Institute of Materials Testing, Berlin, in 1969.

I was awarded the Special Creativity Award of the National Science Foundation in 2005, 1996 and 1988, the 1991 High Polymer Physics Prize of the American Physical Society, and the American Chemical Society Creative Polymer Chemist Award in 1985, and Fellow of American Physical Society, 1986 and Fellow of the American Association for the Advancement of Science (2003).

I have authored or co-authored over 350 publications in academic peer-reviewed journals, and am named as a co-inventor on 12 patents. I am also on several editorial boards of professional journals and serve on the National Research Council's Board on Army Science and Technology.

I received a B.S. in Mechanical Engineering and Engineering Science from the University of Massachusetts in 1969 and a Ph.D. in Materials Science from Cornell University from 1974. My research interests include polymer physics and engineering, the morphology of polymers, especially block copolymers, application of electron microscopy and X-ray techniques to material characterization, optical and mechanical properties of block copolymers and liquid crystalline polymers, the effect of processing on structure-property relationships, interference lithography, photonic and phononic crystals.

2. I am an inventor in the above-reference application. As an inventor, I stand to derive a portion of any financial benefit from licensing this patent application and/or patent issuing therefrom.

3. I have read and am familiar with the above-referenced application, and I have read and am familiar with the outstanding Office Action, dated June 26, 2006, in this case, along with the art cited by the Examiner in the Office Action: Lee et. al., *Macromolecules* **1989**, 22, 2602-2606 ("the Lee reference"), and the claims in the above-referenced application in their latest form.

4. Based on my professional education and experience, and my understanding of the level of ordinary skill in the field of polymer physics and engineering in general and block co-

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polymers in particular, it is my understanding that the porous membranes described in the Lee reference do not include a plurality of periodically occurring separate domains, with at least a first and a second domain each being topologically continuous in all directions. Rather, the porous membranes described in the Lee reference contain discrete domains that are either discontinuous in all directions (spheres), discontinuous in all directions normal to their axis (cylinders), or discontinuous in all directions that are not approximately parallel to their surfaces (lamellae).

5. In support of the conclusion expressed in paragraph 4, above, I note that the Lee reference teaches porous membranes from block copolymers having lamellar, cylindrical, and spherical domains, which are discrete structures. In discrete structures, while domains may occur periodically throughout the structure, the domains are physically isolated from, and not in physical contact with, other like domains in the structure (page 14, lines 8-10 of the specification of the above-referenced application). In contrast, a topologically continuous domain is a domain that is continuous in the sense that a particular domain forms an infinite set of continuous pathways through the structure in all directions (for example, see page 13, line 32 – page 14, line 2 of the specification). The Lee reference describes porous membranes formed from discrete structures, including structures having lamellar, cylindrical, and/or spherical domains, wherein the domains are removed to form voids. Thus, the voids of the porous membranes are physically isolated from other like voids in the structure and do not form a topologically continuous domain.

In the case of a porous membrane formed from the removal of spherical domains, the resulting structure may have spherical voids surrounded by one, topologically continuous domain. However, the porous membrane does not have a first and a second domain each being topologically continuous, as described by the above-referenced application.

6. In further support of the conclusion expressed in paragraphs 4 and 5, above, the Lee reference specifically states that the porous membranes formed from spherical domains show “discontinuous sphere like microphase separation.” The Lee reference also describes porous membranes formed from lamellar structures which exhibit “continuous channels through the membrane” in the sense that a particular channel spans the membrane in one dimension, but does

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not indicate that a topologically continuous domain was formed or that the materials allow for three-dimensional transport in all directions. Rather, in Lee, the porous membranes formed from lamellar structures only allow transport in directions that are parallel to the surfaces of the lamellae. Transport along any direction that is not strictly parallel to the lamellar surfaces (e.g., a direction that has a component along the normal to the lamellar surfaces) is blocked since the transport path is interrupted by an interface between two different, lamellar domains (e.g., the transport path is discontinuous). Similarly, in porous membranes formed from cylindrical structures, where cylindrical channels span the membrane in one dimension, transport is only allowed in a direction that is precisely parallel to the cylinder axes and is blocked in any other direction. By contrast, ONLY three-dimensionally, bicontinuous structures allow transport across a sample in any general direction without having to cross numerous interfaces between the two types of different domains and, therefore, the transport paths are continuous in all directions. This is particularly advantageous in producing nanoporous membranes having high throughput.

To further illustrate this point, a series of exemplary block copolymers, with a variety of microdomain morphologies, is illustrated below in Figure 1. These are provided for purposes of example only, and are not to be interpreted to represent all possible block copolymer morphologies within the scope of the pending claims. Of the block copolymers shown in Figure 1, the only structures that contain at least a first and a second domain, each being topologically continuous (bicontinuous), are the double gyroid and double diamond structures. In the double gyroid and double diamond structures, the darkened, multiple domains (each structure has different domains of different darkness) are topologically continuous along the x, y and z directions, indeed along all directions, and the white domain is also topologically continuous along all directions. In the other morphologies illustrated in Figure 1, the domain continuities are as follows: for spherical morphologies, discrete spheres (darkened domains) exist, as well as a topologically continuous domain (white matrix; continuous in all directions); for cylindrical morphologies, cylinders which are continuous in only one direction along the cylinder axis (darkened domains) exist, as well as a topologically continuous domain (white matrix; continuous in all directions); for lamellar morphologies, lamellae which are continuous in directions strictly parallel to the lamellae surfaces (darkened lamellae) exist, as well as white lamellae (which are also continuous in directions strictly parallel to the lamellae surfaces). Of the structures shown in Figure 1, ONLY the double gyroid and double diamond are

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simultaneously three-dimensionally topologically continuous in a plurality of periodically occurring separate domains (the darkened domains and the white domain). In the Lee reference, the materials, if constructed properly according to the teachings of Lee, *inherently do not have* a plurality of periodically occurring separate domains, with at least a first and a second domain each being topologically continuous, as is recited in pending claim 1 and as is the case for the double gyroid and double diamond illustrative arrangements of Figure 1.

As noted, the materials of Lee are similar or identical to the spherical, cylindrical, and/or lamellar structures illustrated in Figure 1. In the bicontinuous materials of our invention, as recited in claim 1, one is guaranteed to always have sample spanning high transport paths in any direction for optimal transport, which is a significant advantage. Those of ordinary skill in the art would immediately recognize that the materials of Lee would not be expected to have these properties.

As an illustrative example of the material of the invention as recited in claim 1, Figure 2 shows an SEM image of a material that was made from a double gyroid polystyrene-polyisoprene diblock copolymer, with the polystyrene domains forming a topologically connected network and the polyisoprene forming a topologically connected matrix. UV-ozone etching was used to selectively remove the polyisoprene matrix, leaving behind the topologically connected network of polystyrene domains. The polyisoprene matrix was able to be completely removed because this type of domain is completely interconnected to itself (topologically continuous) and the etchant can access all parts of the structure. The remaining, topologically connected network of polystyrene domains is self-supporting because all of the polystyrene domains are interconnected to themselves. Alternatively, a polystyrene selective etchant could be used to selectively remove the polystyrene domains, leaving behind the topologically connected network of polyisoprene domains, since the etchant would be able to access all parts of the topologically connected polystyrene domains. Thus, the resulting materials would contain at least a first and a second domain, each being topologically continuous (e.g., a bicontinuous material), with transport properties that are independent of direction. This is not the case for materials formed from lamellar, cylindrical, and spherical microdomain morphologies.

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Figure 1. Schematic of block copolymer microdomain morphologies.

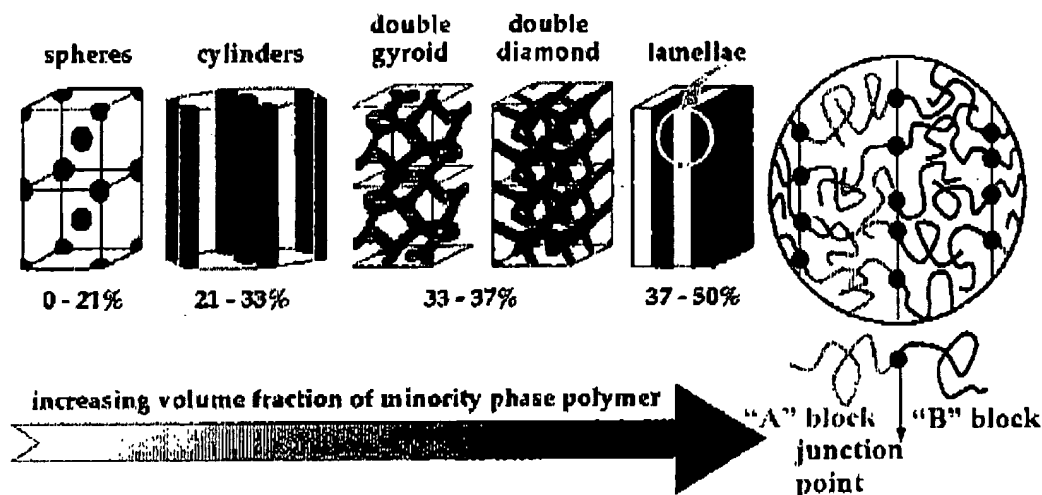
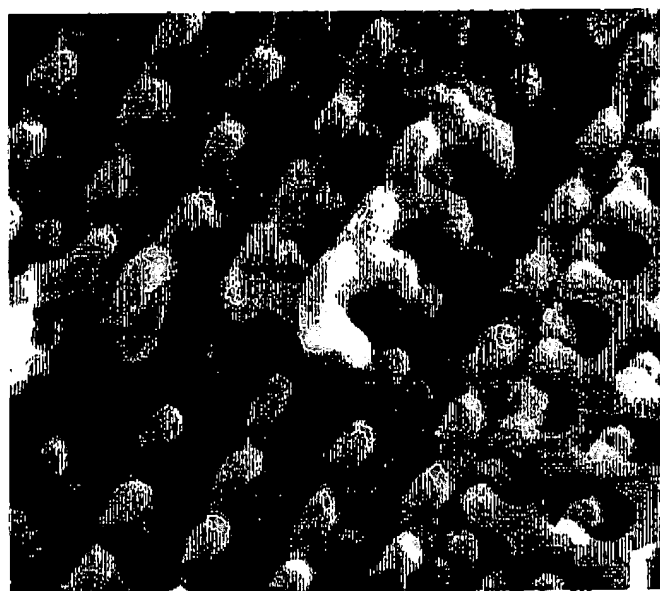
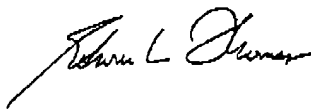


Figure 2. SEM image of an air continuous – polymer continuous structure formed by etching away of the polyisoprene matrix and leaving the topologically interconnected polystyrene domains. The polystyrene is continuous in all directions and the air phase is likewise continuous in all directions. This is a bicontinuous material.



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7. I hereby declare that all statement made herein are of my own knowledge and are true and that all statements made on information and belief are believed to be true, and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of this documents and any patent which may issue from the above-identified patent application.



Prof. Edwin L. Thomas
Department of Materials Science and Engineering
Massachusetts Institute of Technology
Room 35-421
77 Massachusetts Ave
Cambridge MA 02139 USA

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